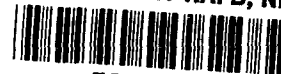NASA TN D-2287

EFFECTS OF INTERSTITIAL IMPURITIES ON THE LOW-TEMPERATURE TENSILE PROPERTIES OF TUNGSTEN

by Joseph R. Stephens

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Cleveland, Ohio

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SUMMARY

A study was undertaken to determine the effects of the interstitial impurities oxygen and carbon on the mechanical properties of polycrystalline tungsten and high-purity tungsten single crystals. Results of tensile tests showed that additions of both oxygen and carbon to polycrystalline tungsten produced a marked increase in the ductile to brittle transition temperature. Oxygen and carbon produced a much smaller increase in the transition temperature of the single-crystal specimens compared with equivalent amounts of impurities in the polycrystalline specimens. Addition of oxygen to polycrystalline tungsten lowered both the ultimate tensile strength and the yield strength, but had no measurable effect on the strength properties of single-crystal specimens. Carbon additions to both polycrystalline and single-crystal specimens did not affect the ultimate tensile strength; however, a large increase in the yield strength resulted. The results suggest that oxygen embrittlement in tungsten is caused by grain-boundary segregation, while carbon embrittlement results from an interaction between carbon atoms and dislocations within the tungsten lattice.

INTRODUCTION

It is generally accepted that interstitial impurities have a detrimental effect on the ductility of the group VI A body-centered-cubic metals. It has been demonstrated that oxygen, carbon, and nitrogen greatly increase the ductile to brittle transition temperature of both chromium and molybdenum (refs. 1 to 7). To date, however, there have been only a limited number of investigations to determine the effects of specific interstitial impurities on the mechanical properties of tungsten (ref. 8). Although few quantitative data are available, it appears that tungsten has a very low solubility for both oxygen and carbon, on the order of several parts per million at room temperature. Since it is not uncommon to have oxygen and carbon present in amounts greater than their solubility limits, grain-boundary segregation of these impurities would be expected to occur.

This study was undertaken to determine the effects of oxygen and carbon on the mechanical properties of both polycrystalline tungsten and high-purity tungsten single crystals. Of special interest was the effect of these impurities on the ductile to brittle transition temperature.

MATERIALS

The material used for this investigation was commercial sintered and swaged 1/8-inch-diameter tungsten rod. High-purity zone-melted tungsten single crystals each with the same orientation were prepared from the same starting material. The major impurities of the starting material are listed in table I.

TABLE I. - MAJOR IMPURITIES IN TUNGSTEN TENSILE SPECIMENS

Material	Element											
	N^a	H^b	O^b	C^c	Mo^d	Fe^d	Ti^d	Al^d	Mn^d	Cr^d	Ni^d	V^d
	Impurity content, ppm											
Poly-crystal-line	10	1	4	8	32	0.2	0.15	0.16	<0.07	0.2	0.85	0.4
Single crystal	8	1	2	4	<1	0.5	<0.05	0.03	<0.07	0.5	0.14	<0.1

[a]Kjeldahl determination, precision, ±5 percent at 15 ppm.

[b]Vacuum extraction; precision, ±20 percent at 5 ppm.

[c]Combustion; precision and accuracy, ±10 percent at 9 ppm.

[d]Emission spectrographic determination; precision and accuracy,
  ±30 percent.

Since the starting material was taken from a particularly pure lot of commercial tungsten, which contained only 4 ppm of oxygen and 8 ppm of carbon, it was possible to increase these impurity levels and maintain the impurity concentrations generally found in commercial tungsten (refs. 9 and 10).

EXPERIMENTAL PROCEDURE

Controlled Addition of Impurities

In order to minimize grain size as a variable in the polycrystalline specimens, all specimens - both those to have controlled amounts of impurities added and those to be evaluated with the impurity level of the commercial material - were annealed for 2 hours at 3700° F prior to introduction of the

impurities. It was thought that this annealing treatment 200° F above the maximum temperature used to add the impurities would stabilize the grain size of all specimens at approximately the same grain size. (As noted later, however, the grain size did increase while oxygen was being added even though the temperature ranged from 3000° to 3500° F.)

Addition of oxygen was accomplished by heating a machined tensile specimen in a sealed tungsten capsule containing tungsten oxide as is shown in figure 1. After loading the capsule, the tungsten plug was inserted and welded in place. Both electron-beam welding in vacuum and heliarc welding in an argon atmosphere were successfully employed for the welding process. Preliminary experiments indicated that a range of oxygen concentration could be achieved by varying the time and temperature at which the capsulated specimens were heated. Table II lists the actual conditions used for adding oxygen to tungsten.
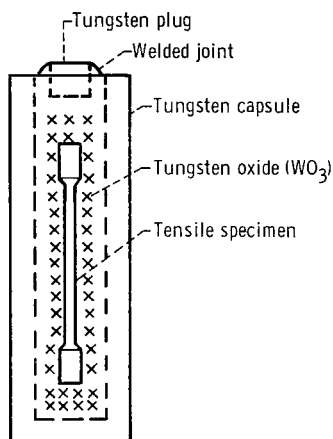
Addition of carbon was performed by heating the specimens in a propane atmosphere in the temperature range from 1500° to 1800° F. The carbon concentration was varied by changing the amount of propane introduced into the furnace. During this low temperature reaction treatment, a thin carbon-rich layer was deposited on the surface of the tensile specimens. Prior to removal from the furnace, the specimens were heated at 2600° F in vacuum for $1\frac{1}{2}$ hours to form an adherent tungsten carbide (W_2C) surface layer. The specimens were subsequently heated to 3500° F in vacuum for 4 hours to allow diffusion of carbon into the specimen.

Figure 1. - Capsule employed to add controlled amounts of oxygen to tungsten.

Since the introduction of oxygen and carbon in the manner described involved diffusion of these impurities into the metal, it was necessary to determine whether the distribution of impurities was uniform across the diameter of the test specimen. Metallographic examination of the specimens containing added amounts of impurities indicated that they all contained second phases distributed uniformly across the diameter of the test specimen. Chemical analyses of the interior of the rod only (after it was machined to about one-half the original diameter), agreed well with similar analyses for the entire cross section of the rod thus indicating that the impurities were uniformly distributed throughout the specimen.

TABLE II. - CONDITIONS EMPLOYED FOR ADDING

OXYGEN TO TUNGSTEN

Time, hr	Temperature, °F	Oxygen content, ppm
5	3000	10
3	3400	30
15	3500	50

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Specimen Preparation and Testing

Tensile tests were conducted on buttonhead tensile specimens machined from both the 1/8-inch-diameter commercial rod and the zone-melted single crystals. As noted previously, the machined specimens were vacuum annealed for 2 hours at 3700° F. Prior to testing, all specimens were electropolished from the 0.080-inch ground diameter to a 0.070-inch diameter to remove surface scratches that resulted from grinding the tensile specimens and also from surface contamination that might have occurred on the specimens with controlled impurity addition. Tensile tests were performed in vacuum (<5 μ) at a constant crosshead speed of 0.005 inch per minute past the yield point and then at 0.05 inch per minute to failure.

RESULTS AND DISCUSSION

Oxygen Additions

The effects of oxygen additions on the ductile to brittle transition temperature of recrystallized tungsten specimens are shown in figure 2. The
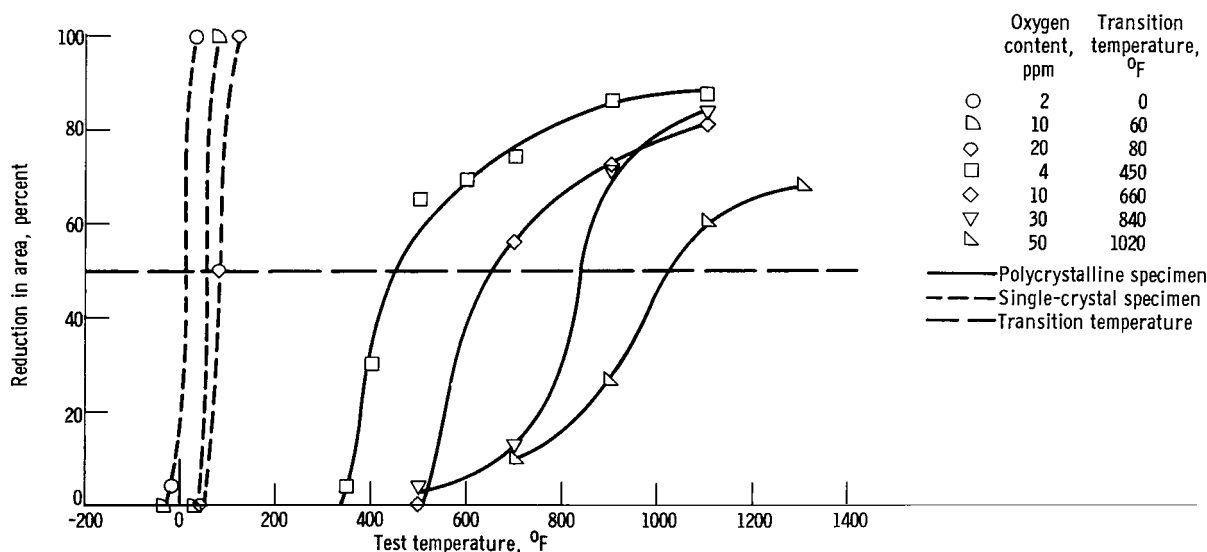
Figure 2. - Effect of oxygen on ductility of tungsten.

transition temperature is arbitrarily defined as that temperature at which 50 percent reduction in area would occur. The recrystallized commercially pure specimens, analyzed to contain 4 ppm of oxygen, had a transition temperature of 450° F. Additions of oxygen to raise the total content to 10, 30, and 50 ppm oxygen (0.0115, 0.0345, and 0.0575 atom percent) increased the transition temperature to 660°, 840°, and 1020° F, respectively.

Figure 3 shows a typical microstructure of the polycrystalline material containing 50 ppm oxygen where a second phase is evident at the grain boundaries. The grain size of the specimens increased from 0.05 to 0.12 millimeter

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when the oxygen content was increased from 4 to 50 ppm. Figure 4 shows a typical brittle fracture of a specimen containing oxygen as the added impurity. It can be seen that the brittle fracture is intergranular, which is the type normally encountered in recrystallized polycrystalline tungsten (ref. 11).
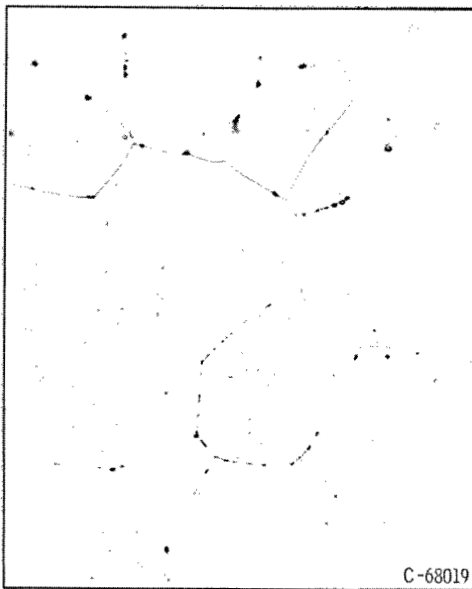

Figure 3. - Microstructure of polycrystalline tungsten. Oxygen content, 50 ppm. X150.
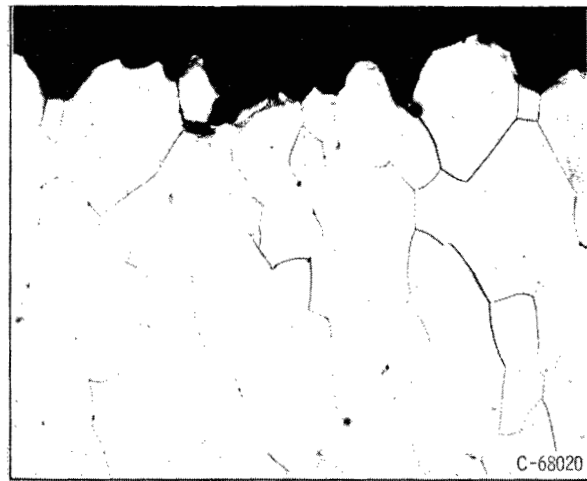

Figure 4. - Microstructure of polycrystalline tungsten showing brittle intergranular fracture. Test temperature, 700° F; oxygen content, 50 ppm. X150.

The effect of oxygen additions on the ductile to brittle transition temperature of the single-crystal specimens is also shown in figure 2, where a comparison can be made with the effect of oxygen on the ductility of polycrystalline tungsten. The effect of oxygen on the transition temperature of single-crystal specimens, as shown in this figure, is much less than the effect of similar additions to polycrystalline tungsten. The zone-melted single crystals, which contained 2 ppm of oxygen (0.0023 atom percent) had a transi-
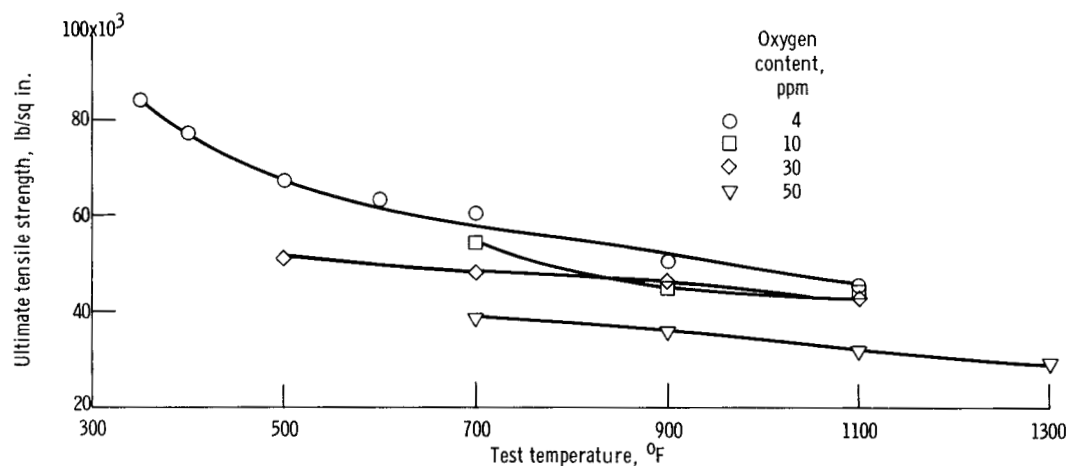

Figure 5. - Effect of oxygen on ultimate tensile strength of polycrystalline tungsten.

tion temperature of 0° F. Increasing the oxygen contents to 10 and 20 ppm (0.0115 and 0.0230 atom percent) increased the transition temperature to 60° and 80° F, respectively. Comparing this to the polycrystalline material indicates that 10 ppm of oxygen increased the transition temperature more than 200° F, and that 30 ppm produced an increase of approximately 400° F. These data strongly suggest that segregation of oxygen at grain boundaries of tungsten is the main cause of the resulting embrittlement.

Figure 5 shows the effect of oxygen additions on the ultimate tensile strength of the polycrystalline material. Increasing amounts of oxygen produced a progressive lowering of the ultimate tensile strength. Tungsten containing an oxygen concentration of 50 ppm had an ultimate tensile strength approximately 30 percent lower than that of the commercial rod.

Yield strengths were similarly lowered with increasing oxygen content as shown in figure 6. The 50-ppm oxygen content reduced the 0.2-percent yield strength approximately 50 percent from that of the commercial rod containing
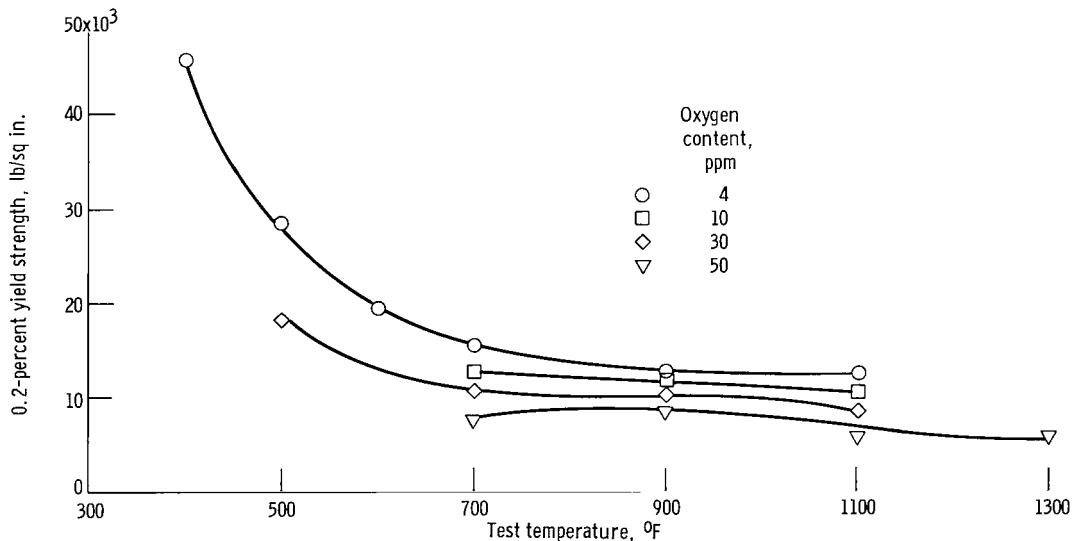

Figure 6. - Effect of oxygen on yield strength of polycrystalline tungsten.

4 ppm of oxygen. This lowering of the yield strength of the polycrystalline material is believed to be due to the removal of carbon and/or other impurities from the starting material on addition of oxygen to the test specimens. Because of the limited accuracy of chemical analyses for carbon and other impurities at these low levels, this theory cannot be verified. Oxygen additions to single-crystal specimens did not change the ultimate tensile strength or yield strength from the strengths of zone-melted crystals.

The observed embrittlement of tungsten by oxygen is explained by the theory for brittle fracture developed in reference 12 where the propagation of cracks is considered. The theory predicts a ductile to brittle transition. The following criterion defines this transition point:

$$\sigma_y k_y \ d^{1/2} \geq \beta \mu \gamma \tag{1}$$

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where σ_y is the yield stress, k_y is a measure of the unpinning stress, 2d is the grain diameter, β, a constant, is unity for uniaxial tension, μ is the modulus of rigidity, and γ is the effective surface energy for fracture. When the left side of the equation exceeds the right side under some stress, the crack will grow into a full fracture without plastic flow. Therefore, increasing the terms on the left or decreasing the terms on the right will promote a brittle fracture and thus raise the transition temperature.

The present investigation was not designed to determine all the terms in equation (1); however, a qualitative analysis of the embrittlement of polycrystalline tungsten by oxygen additions may be made by using this theory. When the addition of oxygen to polycrystalline tungsten was made, the grain size was observed to increase and the yield strength to decrease. The net effect was that the quantity $\sigma_y d^{1/2}$ decreased with increasing amounts of oxygen as summarized in table III and shown in figure 7. From equation (1) this

TABLE III. – DATA FOR DETERMINATION OF $\sigma_y d^{1/2}$ AND RESULTING

TRANSITION TEMPERATURES FOR OXYGEN AND CARBON

ADDITIONS TO POLYCRYSTALLINE TUNGSTEN

Impurity	Impurity content, ppm	Grain diameter, 2d, mm	$d^{1/2}$, $mm^{1/2}$	$d^{1/2}$, $in.^{1/2}$	Yield strength (σ_y) at 750° F, lb/sq in.	$\sigma_y d^{1/2}$, lb-in.$^{3/2}$	Transition temperature, °F
Oxygen	4	0.05	0.224	0.0444	14,500	644	450
	10	.05	.224	.0444	12,500	556	660
	30	.06	.245	.0485	11,000	534	840
	50	.12	.347	.0688	7,800	537	1020
Carbon	8	0.05	0.224	0.0444	14,500	644	450
	36	.05	.224	.0444	26,100	1160	695
	45	.05	.224	.0444	27,500	1220	735
	60	.05	.224	.0444	32,100	1430	780

decrease would tend to lower the ductile to brittle transition temperature. The effect of oxygen on k_y could not be evaluated; however, the absence of a yield point upon adding oxygen to tungsten suggests that k_y was not increased substantially. From the preceding statements, it is believed that the observed increase in ductile to brittle transition temperatures of tungsten with increasing oxygen additions (fig. 7) cannot be accounted for by the terms on the left side of equation (1).

Based on this premise, the more brittle behavior with increasing oxygen content must be due to a decrease in the right side of the equation or more specifically to a decrease in the surface energy for brittle fracture. However, a determination of the magnitude of this term is not afforded by the available data. It is noteworthy that the amount of grain-boundary precipitate increases significantly with increasing oxygen content. It is believed that

the observed increase in ductile to brittle transition temperature with increasing oxygen content arises from the segregation of oxygen at grain boundaries, which progressively lowers the surface energy for crack propagation. All fractures below the transition temperature were intergranular. A similar behavior has been observed in iron (ref. 13) where segregation of oxygen at grain boundaries increased the transition temperature. Increasing the oxygen content changed the fracture path in iron from transgranular to intergranular.

In zone-melted single crystals of tungsten (no grain boundaries), additions of oxygen resulted in only a minor increase in the transition temperature further supporting the idea that grain-boundary segregation of oxygen in tungsten is the main mechanism of embrittlement.

Carbon Additions

The effect of carbon on the ductile to brittle transition temperature of polycrystalline tungsten is shown in figure 8. As with oxygen additions, an increase in transition temperature with increasing amounts of carbon was observed. The transition temperature of the commercial rod having a carbon content of 8 ppm
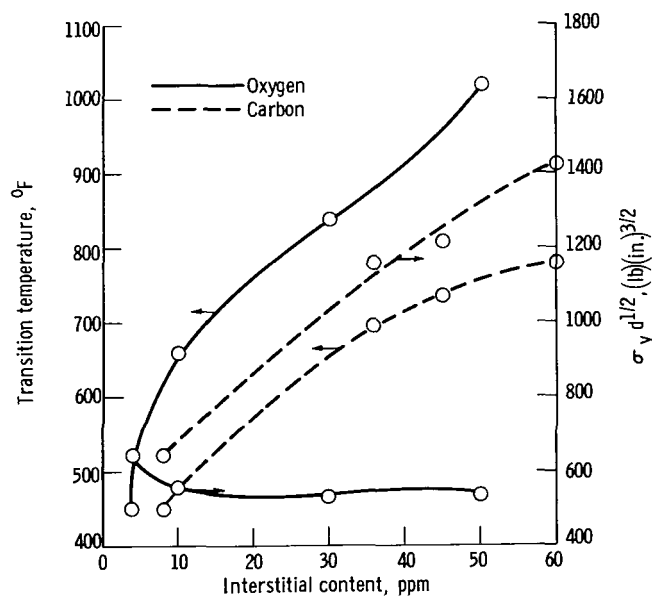

Figure 7. - Comparison of changes in transition temperature and $\sigma_y d^{1/2}$ at 750° F as functions of interstitial impurity content.

(0.0121 atom percent) was 450° F. Increasing the carbon contents to 36, 45, and 60 ppm (0.0552, 0.0690, and 0.0919 atom percent) increased the transition temperature to 695°, 735°, and 780° F, respectively. Although this is a substantial increase in the transition temperature, it is not as great as that observed for similar oxygen concentrations.
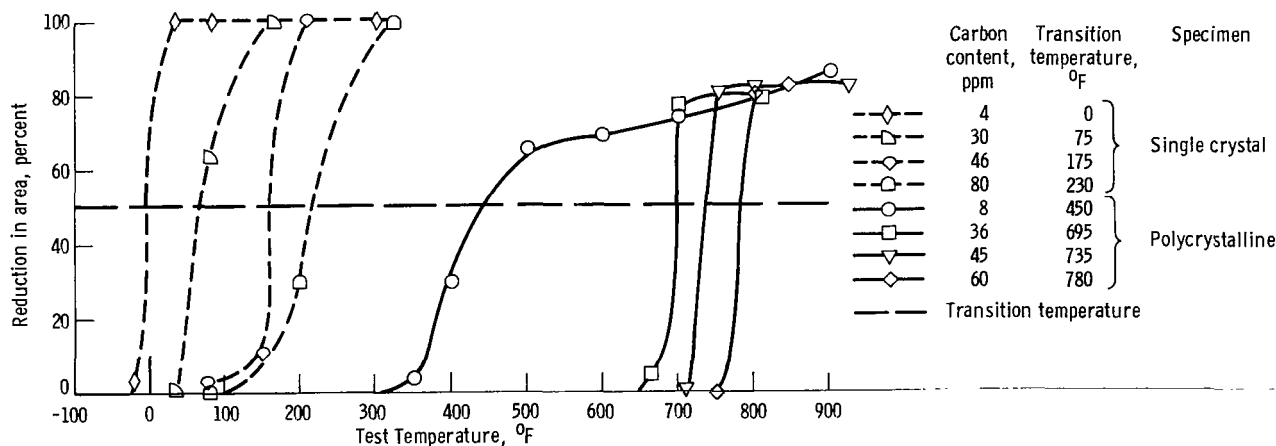

Figure 8. - Effect of carbon on ductility of tungsten.

The effect of carbon on the ultimate tensile strength of polycrystalline tungsten is shown in figure 9. The curve is drawn through the values for the recrystallized starting material. It can be seen that carbon additions did not affect the ultimate tensile strength of the polycrystalline specimens since the data for the specimens containing the original carbon contents and those with added carbon appear to fall on the same curve of ultimate tensile strength as a function of test temperature.
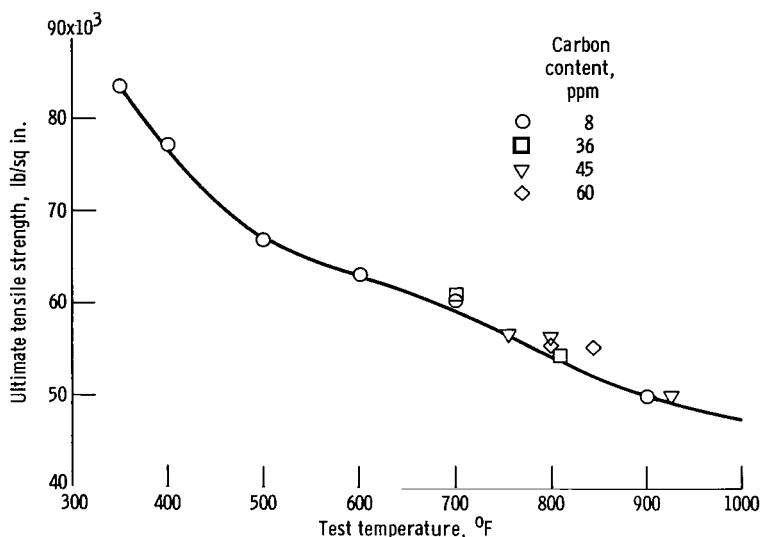
Figure 9. – Ultimate tensile strength of polycrystalline tungsten with varying carbon content.

The effect of carbon on the yield strength of polycrystalline tungsten as a function of temperature is shown in figure 10. Carbon additions at these levels produced a yield point drop in the load extension curve; therefore, the values plotted for the specimens containing 36, 45, and 60 ppm of carbon

are what is normally called the lower yield point. It can be seen that increasing the carbon content from 8 to 36 ppm results in approximately a twofold increase in the yield strength. Increasing the carbon content to higher levels resulted in only a slight increase in the yield strength from that of the 36-ppm carbon level.
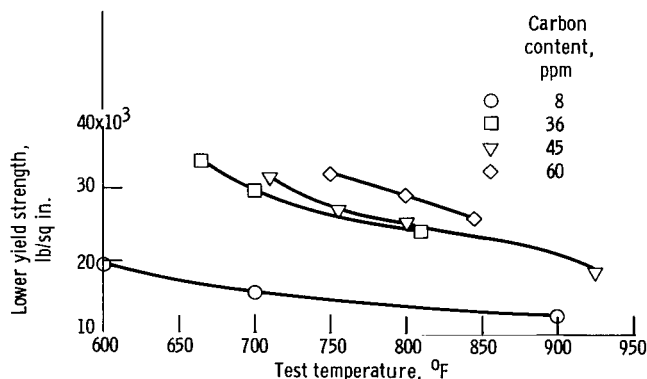
Figure 10. – Temperature dependence of yield strength in polycrystalline tungsten with varying carbon content.

Tungsten with carbon in the range of 36 to 60 ppm contained a second phase at the grain boundaries, as shown in figure 11. The brittle fracture of these specimens was primarily transgranular, as shown in figure 12. This is in direct contrast with the commercial starting material and with specimens to which oxygen had been added, where the brittle fracture was observed to be intergranular.

The effect of carbon additions on the ductile to brittle transition temperature of zone-melted single crystals is shown in figure 8, where a comparison can be made with the polycrystalline specimens that have controlled amounts of carbon added. The transition temperature is increased from 0° F for the zone-melted single crystals containing 4 ppm of carbon (0.0061 atom

percent) to values of 75°, 175°, and 230° F for carbon contents of 30, 46, and
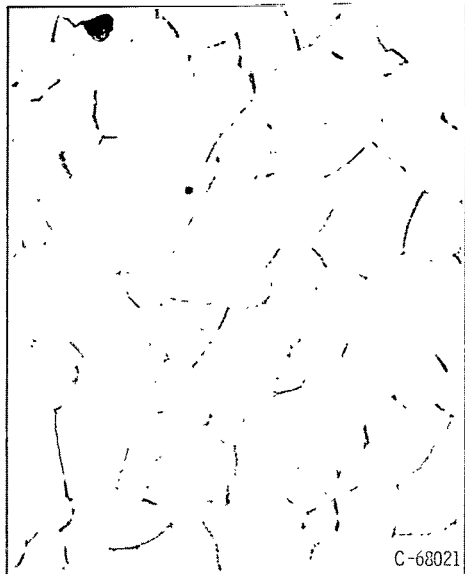80 ppm (0.0460, 0.0705, and 0.1224 atom percent), respectively. The resulting

Figure 11. - Microstructure of polycrystalline tungsten.
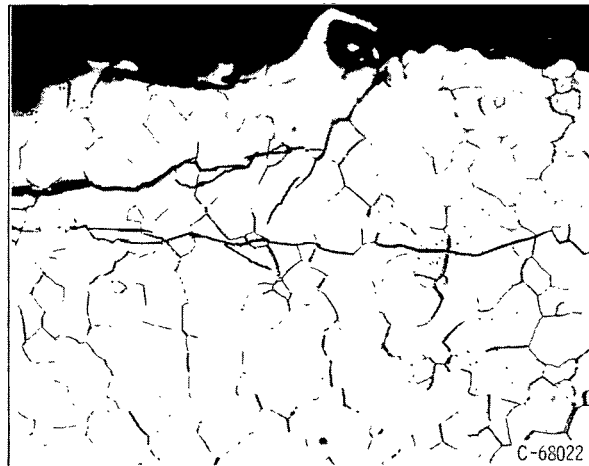Carbon content, 45 ppm. X150.

Figure 12. - Microstructure of polycrystalline tungsten showing
brittle transgranular fracture. Test temperature, 600° F;
carbon content, 45 ppm. X150.

embrittlement of the single-crystal specimens from the increase in carbon con-
tent was not as great as that for similar levels of carbon added to the poly-
crystalline specimens. For example, 46 ppm of carbon produced an increase of
175° F in the transition temperature of the single-crystal specimens, while
45 ppm carbon produced an increase of 285° F in the transition temperature of
the polycrystalline material. This behavior is similar to the results ob-
tained for the single-crystal and polycrystalline specimens containing oxygen
as the embrittling impurity.
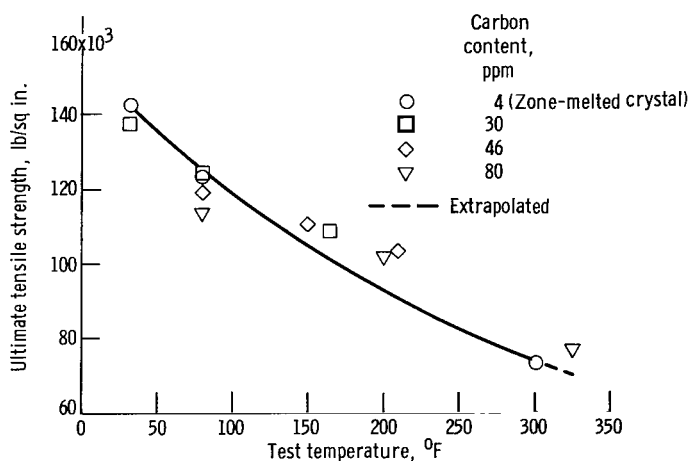
Figure 13. - Effect of carbon on ultimate tensile strength of tungsten single
crystals.

Carbon added to the
single-crystal specimens did
not significantly affect the
ultimate tensile strength, as
shown in figure 13. This was
also true for the polycrystal-
line specimens shown in fig-
ure 9.

Figure 14 illustrates the
effect of carbon on the yield
strength of the single crystals
as a function of temperature.
Both upper and lower yield
strengths as a function of test
temperature are shown compared
with the 0.2-percent offset
yield strength of the unal-
loyed single crystals that ex-

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hibited smooth load-deformation curves. It is of interest to note that the upper and lower yield strengths are independent of carbon content at these carbon levels (30 to 80 ppm of carbon), that is, all of the upper yield strengths fell on one curve and all the lower yield strengths fell on another curve, regardless of carbon content. The upper yield strengths are approximately double the 0.2-percent offset yield strength of the unalloyed single-crystal specimens. The yield point drop was approximately 15,000 pounds per square inch over the temperature range investigated.

Considering again the terms in equation (1) for brittle fracture, some insight may be gained into the operating mechanism for carbon embrittlement of polycrystalline tungsten. As determined experimentally the terms on the left side of the equation were changed in the following manner: the yield stress was increased with increasing carbon content, and no change was detected in the grain size of the specimens containing additional amounts of carbon from that of the starting material (0.05-mm grain diameter). Therefore, the net effect on the quantity σ_y $d^{1/2}$ was to increase with increasing carbon content as listed in table III and shown in figure 7.

As a result of the increase in yield stress and the presence of the yield point at the higher carbon levels, k_y would also be expected to increase from its original value for the 8-ppm carbon content. A quantitative measure of the locking stress k_y is not easily afforded from these experiments; however, as discussed previously (ref. 12) an increase in k_y may be the primary factor in determining the transition from ductile to brittle behavior.

Again a determination of the magnitude of the surface energy for brittle fracture is not afforded by the available data. It is of interest, however, that despite the presence of the second phase at the grain boundaries for the specimens containing 36 to 60 ppm of carbon (fig. 11) brittle fracture resulted from cleavage or transgranular fracture (fig. 12). This indicates that the value of γ at the grain boundaries was greater than it was on the cleavage plane at the higher (36 to 60 ppm) carbon contents.
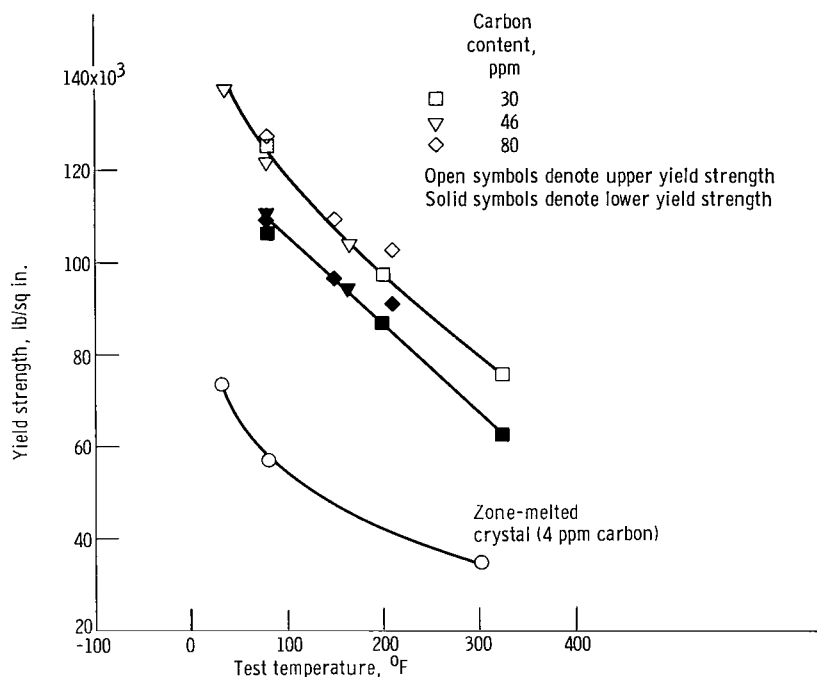
Figure 14. - Effect of carbon on yield strength of tungsten single crystals.

Carbon content, ppm

□ 30
▽ 46
◇ 80

Open symbols denote upper yield strength
Solid symbols denote lower yield strength

Zone-melted crystal (4 ppm carbon)

Yield strength, lb/sq in.

Test temperature, °F

From the previous discussion, it is believed that the observed increase in transition temperature with increasing carbon content (fig. 7) must arise from the observed increase in yield stress and the expected increase in the unpinning or locking stress k_y.

The difference in the magnitude of embrittlement of the polycrystalline and single-crystal specimens may possibly arise from the absence of grain boundaries. As previously suggested (ref. 14), grain boundaries cause forced slip to occur near their vicinity and thus cause a higher stress for the passage of a Luders band through a polycrystalline material than through a single-crystal specimen. This higher stress, in turn, adds to the embrittlement of polycrystalline specimens by increasing the lower yield stress, which will, in turn, increase the left side of equation (1) and lead to brittle fracture. Also, the relatively uniform distribution of the second phase in the single crystals, as opposed to the carbide precipitates being primarily at grain boundaries in polycrystalline specimens, may affect the transition from ductile to brittle behavior.

SUMMARY OF RESULTS

Table IV summarizes the observed results of this investigation for the polycrystalline material. Both oxygen and carbon produced a marked increase in the ductile to brittle transition temperature of polycrystalline tungsten.

TABLE IV. - OBSERVED EFFECTS OF INCREASED OXYGEN AND CARBON CONTENTS ON THE

MECHANICAL PROPERTIES OF POLYCRYSTALLINE TUNGSTEN

Impurity added	Effect on -				
	Transition temperature	Ultimate tensile strength	Yield strength	Fracture characteristics below transition temperature	Microstructure
Oxygen	Increased	Decreased	Decreased	Intergranular	Second phase at grain boundary
Carbon	Increased	No change	Increased	Transgranular	Second phase at grain boundary

Oxygen lowers both the ultimate tensile strength and the yield strength and promotes intergranular fracture. The low solubility of oxygen in tungsten is apparent from the presence of the second phase that is readily observed at concentrations as low as 30 ppm. On the other hand, carbon additions did not produce a change in the ultimate tensile strength but produced a twofold increase in the yield strength when the carbon content was increased from 8 to 36 ppm. Further additions of carbon produced only a slight increase in yield strength over that for the specimens containing 36 ppm of carbon. The brittle

fracture was primarily transgranular in specimens to which carbon had been added. The low solubility of carbon was evidenced by the presence of the second phase that is readily observed at a concentration of 36 ppm.

The results obtained with single crystals showed that, in the absence of grain boundaries, oxygen has only a minor effect on the ductile to brittle transition temperature of tungsten and no measurable effect on the strength properties. Carbon in single-crystal specimens produced a much smaller embrittling effect than it did in the polycrystalline material. Although the ultimate tensile strength was not dependent on carbon content, the yield strength did show a marked increase as the carbon content was increased from 4 to 30 ppm. The upper and lower yield strengths were independent of carbon content between 30 and 80 ppm. The increase in yield strength was similar to the results for the polycrystalline specimens containing carbon.

From these data two different mechanisms of embrittlement are postulated. The resulting embrittlement arising from oxygen addition is believed to be due to segregation of oxygen at grain boundaries, which provides an easy path for intergranular fracture by lowering the surface energy for fracture. Carbon embrittlement is believed to be primarily due to an interaction between dislocations and carbon, which leads to embrittlement by increasing the yield stress and the unpinning stress.

Lewis Research Center
    National Aeronautics and Space Administration
        Cleveland, Ohio, February 6, 1964

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